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RESEARCH ARTICLE

THE INFLUENCE OF PROCESS PARAMETERS ON THE PROPERTIES OF GLASS FOAM PRODUCED FROM INDUSTRIAL WASTE GLASS USING SIC AS THE FOAMING AGENT

A. Kadir SARI ^{1, 2,} ^(b) Recep AKDENIZ ³ ^(b), Ali Murat SOYDAN ⁴ ^(b), Ömer YIDIZ ⁵ ^(b)

1 Gebze Technical University, Institute of Nanotechnology, Department of Nanocience and Nanoengineering, TR-41400 Gebze/Kocaeli, Turkey

2 Vefa Prefabricated Structures Industry Trade and Inc., TR-41455 Dilovası/Kocaeli, Turkey

3 School of Chemical Engineering, University of Birmingham, Edgbaston, Birmingham B15 2TT, UK

4 Gebze Technical University, Institute of Energy Technologies, TR-41400 Gebze/Kocaeli, Turkey

5 Kocaeli University, Faculty of Engineering, Department of Metallurgical and Materials Engineering, TR-41380 Kocaeli, Turkey

ABSTRACT

Glass foams having a low bulk density, 0.22-0.36 g.cm⁻³ were produced using waste glass as the raw material and SiC as the foaming agent. The produced glass foam has very high insulation performance with a very low thermal conductivity of 0.045-0.065 W.m⁻¹.K⁻¹, making it suitable for thermal and acoustic applications. Despite its highly porous structure, it exhibits a very high compressive strength between 0.08-6.99 MPa and good dimensional stability. The environmentally friendly glass foam produced in this study has the potential as a structural and insulation material in construction applications. Nano-sized foaming agents enhance the efficiency of the reaction in the manufacturing process. The properties such as volume change, weight loss, microstructure, mineralogical structure, sintered bulk density, and porosity of the prepared samples were measured and analyzed. The characterization of the thermal and the mechanical properties of the samples was performed by testing their compressive strength, and thermal conductivity.

Keywords: Insulation materials, Glass foam, Waste glass, Foaming agent, SiC

1. INTRODUCTION

Glass foam is an important heat and acoustic insulation material with high porosity, low bulk density, low heat transfer coefficient, waterproof and non-flammable properties [1-9]. They are usually classified by their bulk density (~0.1 g.cm⁻³) due to their porous microstructure and hence lightweight [1, 3, 4,10-12]. Glass foams have relatively high mechanical strength, chemical and dimensional stability, and low water absorption due to the closed porosity [1, 3, 4, 11-13] when they were compared to other insulation materials. They are biologically, thermally, and chemically inert materials, not affected by many environmental effects, and do not deteriorate because glass is a natural material [5, 14-17]. There is no linear relationship between density and the water absorption properties, however, the type and concentration of the foaming agent and the pore structure have a very significant effect on the water absorption values of the glass foams [1, 3, 14, 18-20]. All these properties depend on the microstructure of the glass foams (the size of the pores, the alignment, and the thickness of the glass walls between the pores) and their relationship to the density.

Sintering begins at a certain temperature above the softening temperature of the glass, resulting in gas emissions at the molten glass state and thus the foaming and pores are formed by the foaming agents. The structure of these pores and the properties of the glass foams depend on the type of foaming agents, the concentration, and the process conditions [3,21-23]. Many studies were performed in the past to investigate the relationship between the microstructure of the products (the size and distribution

*Corresponding Author: <u>asoydan@gtu.edu.tr</u>

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of pores, the bulk density, etc.) [1-27]. Various raw materials, wastes, forming agents, and the processing parameters (temperature, time, ramp rates) were studied to determine the suitable wastes and to increase the usage of waste in the production of foams [1-27].

The glass foams produced by using carbonates as "neutralizing" foaming agents cause CO₂ gas outflow and therefore have larger pores and higher water absorption values (50-70%) [3,5]. However, they show high performance in terms of sound insulation. The glass foams produced with carbon-containing "reductive" foaming agents such as coke, soot, graphite, and SiC, have low water absorption values (10-15%) and mostly closed pores were formed in the microstructure [3, 5, 28]. For this reason, they are preferred in the production of glass foams for heat insulation. The reason for the gas formation and the dissolution is due to the oxidation of the blowing agents of molten glass-dissolved gases, mostly oxygen and sulfuric anhydride [3]. To obtain the desired microstructure and good thermal insulation properties, the glass powder is expected to exhibit high oxidation potential, and no tendency to crystallize. With this purpose in mind, glass powder should be ground to have a specific surface area of at least 1500 m²/kg. The surface tension between the glass viscosity and the foaming temperature must be low [29]. The reaction temperature is generally higher than about 1000 °C when compared to other compounds such as carbonates, carbon, organic compounds. According to DTA / TGA results of pure SiC, reactions (1) and (2) take place above 900 °C;

$$\begin{aligned} \operatorname{SiC} + 2\operatorname{O}_2 &\to \operatorname{SiO}_2 + \operatorname{CO}_2 \\ \operatorname{SiC} + \operatorname{O}_2 &\to \operatorname{SiO} + \operatorname{CO} \end{aligned} \tag{1}$$

$$31C + O_2 \rightarrow 310 + CO$$

Another possible reaction is;

$$\mathrm{SiC} + \mathrm{SiO}_2 \to \mathrm{CO}_2 + 2\mathrm{Si} \tag{3}$$

Foaming agents enable the formation of numerous small and closed pores and the separation of glass particles [3]. SiC and Si₃N₄ are typical foaming agents in the production of borosilicate-based glass foam in a wide temperature range [2]. The size and structure of the pores were possible to modify by the amount of SiC, Si₃N₄, and oxides of transition metals [2]. For the waste glasses with 4-5 wt% of SiC and TiN, the pores were small and completely bonded to one another and the TiN-doped samples had a double-dispersed microstructure. The resulting bending and compression strengths were 4-68 MPa and 4-267 MPa, respectively, depending on the porosity [21]. The glass foams with a bulk density of 8% (containing 92% porosity) were produced from the mixture (<37 µm sized) prepared by using the soda-lime waste glass, SiC waste used in the polishing of the glass, and MnO₂ by heat treatment at 950 °C [14]. The foaming of the waste glass powders was observed to start during the glazing of the soda-lime glasses at 670-680 °C while the heat treatment range was 700-950 °C. The products have the maximum volume expansion (600-700%) and low bulk density (0.21-0.38 g/cm³) at these temperatures (670-680 °C) [18]. This expansion was thought to be caused by the disintegration of organic compounds in the oil-based refrigerant on the surface of the polished glass [18]. However, it should be noted that the decomposition temperatures of the organic components are very low. The pore distribution in the microstructure of the produced glass foam is not homogenous in the conditions at 700-900 °C, using an admixture of the lead silicate glass and 1-7 wt% TiN or SiC. Pb (II) in the glass foam was reduced to Pb (0) with the increase in the amount of TiN or SiC [22]. The microstructure of the glass foam produced at 750-950 °C using Pb-containing waste cathode ray tube glass and 1-9 wt% TiN or SiC has been observed to have a bigger pore size due to an increase in the reaction time, the sintering temperature, and the amount of reducing agent [23]. It was reported that there is a direct linear relationship between the bulk density and the fracture strength of the glass foam depending on the heating rate, the hold time, and the foaming effect. These properties could be optimized with the duration of hold time and the amount of foaming agent [4].

In this study, we have investigated the effect of the process parameters on the properties of the glass foam produced from the industrial waste glass using silicon carbide (SiC) as the foaming agent. The volume change, the weight loss, the microstructure, the mineralogical structure, the compressive strength, the bulk density, the porosity, and the thermal conductivity of the produced glass foam samples were all measured and analyzed.

2. EXPERIMENTAL PROCEDURES

Experimental studies on glass foam production were performed at Gebze Technical University (GTU) laboratories.

2.1. Materials

The waste glass powders as raw materials from Cam Kirigi Co. Turkey, silicon carbide (SiC) nanopowders as the foaming agents having a purity of 99.5% with grayish white color, also has an average powder size of 202 nm from Company of Nanografi Turkey, and carboxymethylcellulose (CMC) as a binder in form of white powder or fibers having a density of 1.45 g/cm³ from Organik Kimya Co., Turkey were used to produce the foam glass products.

2.2. Production of Glass Foam Materials

The sample codes/names are given according to the mixing ratios of the SiC, heat treatment temperature, and hold time of samples given as a study plan in Table 1. For example; the H2 sample represents the product of firing at 800 °C for 2 hours with 2 wt% SiC content and compacted by a compaction pressure of 5, 10, and 15 MPa, and they were named as H2-5, H2-10, and H2-15 respectively. The heating rate of the furnace for all samples was 10 K/min.

Sample Code*	Heat treatment temperature	Hold time	The SiC ratio, compaction pressure (5, 10, and 15 MPa)** and sample codes in samples		
			1 wt%	2 wt%	3 wt%
А	875 °C	No hold	A1**	A2**	A3**
В	850 °C	No hold	B1	B2	B3
С	850 °C	15 min	C1	C2	C3
D	850 °C	30 min	D1	D2	D3
E	825 °C	30 min	E1	E2	E3
F	825 °C	60 min	F1	F2	F3
G	825 °C	120 min	G1	G2	G3
Н	800 °C	60 min	H1	H2	Н3
J	800 °C	120 min	J1	J2	J3
K	800 °C	180 min	K1	K2	K3
L	775 °C	120 min	L1	L2	L3
М	775 °C	180 min	M1	M2	M3

Table 1: The definition of the glass foam samples made under different production conditions

* 3 samples were produced for each glass foam product (Namely; 27 samples were produced for each product group.). ** Sample code: for example; sample A1 which was prepared by addition of 1 wt% SiC was named as A1-5 for the sample compacted by a pressure of 5 MPa and named as A1-15 for 15 MPa. The same rule was applied also to all samples: M2-10 having 2 wt% SiC and compacted by a pressure of 10 MPa.

The glass parts collected from the waste were broken by a crusher and then ground by a ball mill to the desired grain size. Pre-determined SiC was added to the milled glass powders. The glass and SiC were mixed for 3 hours and a homogeneous mixture was formed. Then, 7 wt% binder was added to moisten the dry mixture. The main purpose is to impart wet strength to the green body. The CMC binder solution was prepared by adding 20 g of CMC to 1 liter of water and stirring for 30 minutes. Subsequently, the mixture of SiC and glass powder moistened with binder was granulated through a

50 μ m sieve to obtain a homogeneous mixture. The granulated powders were pressed at pressures ranging from 5 to 15 MPa by using the stainless-steel mold pressing equipment to obtain the compacted green pellets with a diameter of 30 mm. The pellets were removed from the mold and the specimens were heat-treated at 775-875 °C with different hold times (0-180 min.) in the oven, then were allowed to cool down by themselves.

2.3. Characterization of Produced Glass Foam Materials

The microstructures of the glass foam pellets were investigated by Philips XL30 SFEG scanning electron microscope (SEM). Archimedes method was used by means of the following equation: $\rho_b =$ $\frac{W_a}{W_c - W_b} \cdot D$, to investigate the sinterability and the densification by determining the open porosity and the relative bulk density of the compacted green and sintered bodies of the produced glass foams. Here, ρ_b is bulk density of the sample, W_a is the normal dry weight of the sample, W_b is the weight of the samples in the water after boiling in water, W_c is the wet weight of the samples (in the air) after boiling in water, and D is the density of the distilled water. Compressive strength tests were performed using an Instron 5569 test machine on the five different examples for each sample to determine the mechanical strength of the produced glass foam depending on the foaming agent, the heat treatment temperature, and the porosity volume (bulk density). The dimensions of the samples were 20 mm x 20 mm x 30 mm. The compressive strength (σ_c) is calculated using the following equation: $\sigma_c = \frac{F_i}{A}$ where F_i and A are the instantaneous compressive stress and the cross-section of the samples, respectively. The crystal structure of the raw materials and the trace of the crystalline phase in the glass foam products were determined by X-ray diffraction (XRD) on Rigaku Dmax 2200 Diffractometer using Cu-K_{α} radiation with a characteristic wavelength of $\lambda = 1.5406$ Å ($2\theta = 10-70^{\circ}$) at room temperature. Thermal conductivity measurements were performed with the device of C-Therm Technologies TCI-2-A at room temperature and 0-10 W/(m·K) range. Dimensions for thermal conductivity measurements; cylindrical samples with a diameter of 25 mm and a height of 5 mm were prepared. The properties of the glass foam products were discussed in the results and discussion section.

3. RESULTS AND DISCUSSION

Several experimental studies were carried out to produce the glass foam by using SiC as a foaming agent in a research project. The production conditions of the glass foam, such as the pressing conditions, the heat treatment temperatures and the hold times of the pellets in a furnace, and the mass ratio of SiC and waste glass raw material were studied. The properties of the glass foam products, such as the bulk density, the thermal conductivity, the microstructure, the porosity property, and the mechanical strength were examined. The grain size and XRD analysis of SiC and waste glass raw materials used in the preparation of all samples were presented in Figures 1 and 2, respectively. The waste glass is in an amorphous form and does not contain any impurities in a crystal structure (Figure 2). The SiC powders are crystalline and pure as shown in Figure 2.

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Figure 1. Particle size analysis of the raw materials, SiC, and waste glass



Figure 2. XRD analysis of the raw materials, (a) SiC and (b) the waste glass

Since numerous experimental studies have been carried out within the scope of the project, it is not possible to handle all the parameters that affect the properties of the produced glass foam independently of each other and to present the properties of the whole samples together in a few graphs simultaneously. Therefore, some parameters of the samples were ignored and various characteristics of the samples were examined by comparing the basic two or three parameters to simplify the interpretation of a large amount of data obtained. The results of the analysis of some samples and SEM microstructure images which are very important with the potential to shed light on the whole studies, were analyzed in detail and presented here.

3.1 Physical Properties of Produced Glass Foam Materials

Firstly, the changes of the bulk density, volume expansion, and mass loss in the structure of all glass foams were investigated independently of the parameters such as the effects of pressing pressure, hold

time of samples in the furnace, and heat treatment temperature of the pellets, and the mass ratio of SiC additive. These results were presented in Fig. 3. While the mass of the heat-treated samples decreased very little (0-3.5%), there was a very large increase in volume expansion (113.6-732.5%) and consequently a significant decrease in bulk densities (53.3-88.1%) as shown in Fig. 3. SiC was degraded by heat treatment. There was a mass loss, albeit a small amount, and a large volume expansion was occurred due to the pores formed, and thus the bulk density was reduced. Depending on the production conditions, it was seen that the decrease of the bulk density in the products mostly was by 60-85% and the increase of the volume expansion was by 300-600% as shown in Figure 3.



Figure 3. Mass, volume, and sinter bulk density changes occurring in the structure of the produced glass foam samples.

Depending on the heat treatment temperature, the bulk density changes in the structure of the glass foams produced are shown in Figure 4. The ellipsoid area on the graph shows the range in the sintered bulk density obtainable depending on the sintering temperature, the hold time, the SiC ratio, and the pressing pressure of the pellets. If the hold time at the sintering temperature is higher than required as an example, 30 min. at 875 °C, 75 min. at 850 °C, 150 min. at 825 °C, 240 min. at 800 °C, 240 min. at 775 °C, the glass foam obtained has a high bulk density, and therefore, the loss of the insulating properties is expected. On the other hand, the effect of hold time during heat treatment on the porosity and bulk density exhibits itself at longer periods at lower temperatures (775 °C for 30-90 min.) and shorter periods at higher temperatures (875 °C for 0-30 min.). Another parameter that must be considered is the pressing pressure of the pellets. If the pellets are pressed under high pressure, the diffusion of oxygen into the pellet, which will provide the oxidation of SiC, will be slow. This situation was determined on the observations made during the the experimental studies. It has been observed that pellets pressed with low pressure have high volume expansion in a very short period of time, but volume expansion of pellets pressed with high pressure occurs too late. On the other hand, it has been determined that the CO_2 gas formed in the pellets pressed with low pressure leaves the structure easily due to the heat treatment conditions and the sample shrinks due to the sinter effect and the shrinkage of the samples pressed under high pressure occurs very slowly and in a long time. It was not possible to present an understandable relation/correlation in these results obtained due to this opposite effect of pressing pressure. Therefore, the hold time at the same heat treatment temperature has to be longer so that the reaction $(SiC + O_2 \rightarrow SiO_2 + CO_2)$ can be completely realized in the structure of all SiC grains in the pellet structure. For this reason, the pellets should not be pressed under high pressure. It will provide no improvement in terms of low density and high insulating properties in the final product. However, to avoid any deformation in the production process, it is sufficient to press under a certain pressure to obtain sufficient green strength. In order to achieve the desired sinter bulk density consistently, a lower sintering temperature should be preferred during heat treatment since the tolerance range at higher temperatures is very narrow. However, a long-term heat treatment at low temperatures (eg, 180 min. at 775 °C) increases the cost, so the sintering temperature should be somewhat higher. Therefore, the heat treatment conditions can be easily optimized over the range of 800-850 °C depending on the duration and other parameters to target any desired sinter bulk density.



Figure 4. The bulk density changes in the structure of the produced glass foams depending on the heat treatment temperature (Hold time and other parameters are ignored.).

When the results obtained by ignoring all other parameters that can affect the glass foam properties in the production process are evaluated, it is noted that there is a linear relationship between the raw densities (sinter bulk density) and the thermal conductivities of the produced glass foams (Fig. 5). According to the results, the sintered bulk densities of the glass foams produced were found to be concentrated in the range of 0.22-0.36 g.cm⁻³ and the thermal conductivity values in the range of 0.045-0.065 W.m⁻¹.K⁻¹. As stated above, these results are presented independently of all other parameters. It is possible to obtain the same or similar results at a different set of production conditions. For example, it is possible to obtain the same or similar results by heat treatment at 875 °C for 15 min. or 800 °C for 120 min.



Figure 5. Linear relationship between sintered bulk densities and thermal conductivities of glass foams produced (All parameters are ignored and considered as a whole.).

Although the prepared samples with different pressing pressures and SiC additive ratios were heattreated under the same conditions, there may be differences in sinter bulk density Figure 6 and thus thermal conductivity values Figure 7. Although the pressing pressure does not have a direct effect on sinter bulk density, it causes a longer heat treatment time as it delays the diffusion of air in the heat

treatment medium into the pellet. Therefore, if the pressing pressure is high when all conditions are the same, the heat treatment time must be greater to obtain the same sinter bulk density value. The effects of the parameters that are effective in the production process on the sinter bulk density Figure 6 and hence thermal conductivity values Figure 7 of the produced glass foams are grouped separately.



Figure 6. Effects of process parameters (SiC content, pressure, temperature, and hold time) on the sinter bulk density of the glass foams produced using 1, 2, and 3 wt% SiC as a foaming agent.

Depending on the process conditions, especially the heat treatment temperature and the duration of the exposure, the degree of softening of the glasses, the wetting of the SiC particles, and the oxidation reaction (SiC + O₂ \rightarrow SiO₂ + CO₂) are changing. If the lack of heat treatment temperature and the hold time is insufficient, the desired low sintered bulk density cannot be obtained because the oxidation is not realized completely (Figure 6). On the other hand, when the heat treatment

temperature and the hold time are high, it is found that the viscosity of the glass decreases, the gas discharge occurs easily, and consequently the condensation increases (Figure 6).

In general, there is a linear relationship between the sinter bulk density and thermal conductivity, therefore the thermal conductivity increases as the sinter bulk density increases (Figs. 5 and 6). However, as shown in Figure 6, there is no one-to-one linear relationship between all sinter bulk density and the thermal conductivity values. Depending on the heat treatment conditions and the foaming agent additive proportions, the porosity of some glass foams specimens is large and has a low sinter bulk density value (Figure 6) resulting from their incorporation into each other. It has been found, however, that the thermal conductivity values of the same samples are much lower (Figure 7). This is because the heat transfer is much lower due to the destruction of the walls between the pores resulting from the association of the pores with each other. Process conditions for producing the glass foams with a low coefficient of thermal conductivity to provide excellent thermal insulation can be predicted based on the results presented. To reach targeted sinter bulk density and thermal conductivity values, the heat treatment temperature can be optimized in the following range of conditions; 800-850 °C, 30-60 min. hold time and 1 wt% SiC foaming agent (Figure 7).



Figure 7. Effects of process parameters (SiC content, pressure, temperature, and hold time) on the thermal conductivity of glass foams produced using 1, 2, and 3 wt% SiC as a foaming agent.

3.2. Microstructural Properties of Produced Glass Foam Materials

Figures 8-10 show that pores are very large (eg 300-500 μ m) in the microstructure of low-density glass foam, the walls between the pores are very thin like a membrane, and the other pores are located above and below the connection points of the three pores. In the microstructure of the glass foams with high density, the pore sizes are small ($\leq 300 \mu$ m), the walls between the pores are thicker, and there are dense vitreous structures below or above the connection points where three pores are connected (Figures 8-10). The size, size distribution, and amount of SiC particles, as well as the heat treatment temperature and the hold time at this temperature, affect the size and size distribution of the pores in the foam glass products. When the results from all the studies were compared, the products with the lowest and highest sinter bulk density were measured at 850 °C, 15 min. hold time, with 2 wt% SiC foaming agent addition and at 850 °C, 0 min. hold time, 3 wt% SiC foaming agent additive (Figure 10). On the other hand, the cristobalite form was observed in very small amounts as a crystalline structure (Figure 11).

The glass foam products fabricated previously from the recycled glass powders using the propyl gallate as a foaming agent have a compressive strength between 0.85-5.92 MPa [12]. The glass foam products obtained by using TiN and SiC as a foaming agents have a compressive strength between 4-267 MPa [21]. These values are much higher than the calculations of compressive strength Méar et al. [21] according to Gibson and Ashby's model [30]. The glass foam products manufactured in our study have a compressive strength between 0.08-6.99 MPa as shown in Figure 12, which were determined according to the standard calculation method. Figure 13 shows the relationship between the compressive strength of the glass foams produced under different conditions. It was determined that the glass foam products have an average compressive strength of around 1.5 MPa and a very different stress-strain property as shown in Figures 12 and 13, respectively.

A very small amount of Cristobalite and SiC phases were observed as a crystalline phase in the glass foam products as shown in Figure 11. Although a desired low sinter bulk density can be achieved at temperatures as low as 850 °C and long hold times, the weak peaks of SiC were observed in the XRD analysis of some of the glass foam products according to heat treatment temperature and duration (Fig. 11). This result means that SiC is not completely converted to SiO₂ (oxidation reaction) because the desired temperature has not been reached and/or no heat treatment has been performed for a sufficient period of time. Therefore, a sufficient temperature (825-850 °C) must be used and a sufficient time (30-60 min.) must be allowed for the complete oxidation of SiC during the production process. However, the temperature should not exceed 850 °C to keep the viscosity and avoid condensation.



Figure 8. The SEM micrographs of the glass foams produced under different process conditions.





Figure 9. The SEM micrographs of the glass foam products; (H3-5): 5 MPa, 3 wt% SiC, 800 °C, 60 min.; (H3-15): 15 MPa, 3 wt% SiC, 800 °C, 60 min.; (K1-5): 5 MPa, 1 wt% SiC, 800 °C, 180 min.; (K3-5): 5 MPa, 3 wt% SiC, 800 °C, 180 min.



Figure 10. The SEM micrographs of the glass foam products; (C2): 10 MPa, 2 wt% SiC, 850 °C, 15 min.; (B3): 15 MPa, 3 wt% SiC, 850 °C, 0 min.

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Figure 11. The XRD analysis of some glass foam products obtained at different temperatures.



Figure 12. The effects of process parameters on the compressive strength of the glass foams produced under different conditions.

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Figure 13. The relationship between the compressive stress and strain of the glass foams produced under different conditions.

4. CONCLUSION

SiC can be used as an effective foaming agent material to produce the glass foams having a low bulk density, 0.22-0.36 g.cm⁻³ and a very low thermal conductivity of 0.045-0.065 W/(m·K), by using the waste glass as raw material. An optimum SiC ratio to obtain desired glass foam properties such as thermal conductivity, the bulk density, and a microstructure with open or closed porosity was determined. The long hold times during heat treatment at high temperatures cause the size of porosities to shrink and the bulk density to increase. In heat treatments at high temperatures (850-875 °C), very short periods of hold time (15-30 min.) have a very important effect. And this effect occurs only in very long periods of time (60-180 min.) at low-temperature (775-800 °C) heat treatments. For this reason, when the heat treatment conditions are optimized, the hold times at high temperatures should be closely controlled. The pressing pressure of the pellets does not directly affect the properties of the glass foams, and to achieve the desired bulk density the time of heat treatment could be extended.

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CONFLICT OF INTEREST

The authors stated that there are no conflicts of interest regarding the publication of this article.

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